

2(PRTS)

10/522495

DT01 Rec'd PCT/PTO 26 JAN 2005

SOLLER SLIT USING LOW DENSITY MATERIALS

FIELD OF THE INVENTION

The present invention relates to X-ray metrology. Specifically, the present invention relates to an absorption element used to control the divergence of a beam of X-rays.

BACKGROUND OF THE INVENTION

In recent years, technology involving high energy radiation, such as X-ray and extreme ultraviolet (EUV) radiation has increased. However, because of the very nature of this type of radiation, it is often difficult to control its divergence. One common optical element used for controlling the divergence of an X-ray beam is called a Soller slit. Soller slits generally comprise a set of parallel, or nearly parallel, plates or blades that limit the divergence of an X-ray beam by simple blocking or absorption of divergent rays, which are not allowed to pass through an open section of the array.

Typically, Soller slits have been made of materials of high density to allow for absorption of divergent X-rays. It has been generally thought that materials of high density, such as dense metals (e.g., molybdenum or brass) needed to be used in Soller slits to provide adequate absorption for the high energy divergent X-rays.

Thus, blades of Soller slits have traditionally been made out of sheets of heavy, or highly absorbing metal. Although these metal sheets can be made extremely thin, the mechanical stability of very thin sheets is not good enough for high precision X-ray work. For example, any curling or rumpling of the sheets, which is common with metals, will result in poor transmission through the Soller slit device, and consequently unpredictable divergence.

Metal-foil Soller slits have been made with relatively thick foils (e.g., on the order of 250 μ m). These metal foil devices yield relatively low transmission efficiencies. Moreover,

the transmission efficiencies of such devices worsens as the required divergence is reduced (i.e., the quality of such devices' outputs becomes worse as their design constraints are made more restrictive).

One attempt at producing a Soller slit X-ray collimator using materials other than heavy metals has been described in European Patent No. EP 0354605 B1, applied for by Philips Corp. on behalf of inventor John Joseph Zola. However, the Soller slit described therein requires expensive ceramic materials processing to build, and is therefore less desirable for commercial applications.

Soller slit devices for collimation of X-rays and other high energy radiation have many important commercial applications. One commercial application that makes use of a Soller slit as an X-ray collimator is X-ray diffractometry. X-ray diffractometry is used for examining powders when a slit-limited X-ray beam impinges on a powder, dirt, pharmaceutical product, or anything in a powder or crystalline form. Some examples of elements measured by way of X-ray diffractometry include pharmaceutical pills, powder within capillaries, and powder between plates. X-ray diffractometry can make use of either transmissive or reflective measurements of incident X-rays.

X-ray diffractometry is the most widely used form of X-ray diffraction in the world. Thus, Soller slits that can be used for X-ray diffractometry are highly desirable for multiple commercial diffractometry applications. Additionally, many devices using traditional Soller slits have already been developed for X-ray diffractometry. Because X-ray diffractometry produces and requires measuring such a weak signal, the diffracted X-ray signal must be measured over a long period of time, typically several hours. Therefore, an increase in transmission efficiency of the X-ray optics (e.g., a Soller slit device) would provide huge commercial advantages, as processing time could be greatly reduced because of the stronger incident radiation, which in turn produces a proportionally stronger diffracted signal.

One problem generally associated with Soller slit devices used for such commercial applications as X-ray diffractometry, however, is that they generally have relatively low transmission efficiencies and large divergence angles. For example, a typical, previously available Soller slit device may have a transmission efficiency of 30% or less. Thus, generally well over half of the X-ray radiation passing through the device is lost and unusable.

for measurements in the application in which the Soller slit is being employed. Additionally, typical divergence angles for previously available Soller slit devices generally range from 0.2° to 0.8°. This typical divergence angle is large, and negatively impacts the Soller slit's ability to effectively collimate x-ray radiation for many important commercial applications such as X-ray diffractometry.

Accordingly, it is desirable to produce blades of a Soller slit from a material that provides adequate absorption of diverging X-rays, without the problems associated with prior devices, such as those made from heavy metals. Specifically, it is desirable to provide a Soller slit device that utilizes materials that do not bend, as is the case with traditional metal blades. Also, it is desirable to provide a Soller slit that makes use of relatively low density materials. Additionally, it is desirable to produce a Soller slit device having blades with a thinner profile than metal blades or metal foil blades, and which consequently provide a better transmission efficiency for the device. It is desirable to provide such an increase in transmission efficiency, while maintaining a low divergence angle. Additionally, it is desirable to provide a Soller slit X-ray collimator that provides the above objectives, while being relatively inexpensive to ensure that commercial advantages are maintained.

Furthermore, it is desirable to provide an X-ray diffractometer, or diffractometry system utilizing a Soller slit using the above-mentioned low density materials. Specifically, it is desirable to provide a system for performing high energy radiation diffractometry, which makes use of an increased transmission efficiency of such a Soller slit, and its low divergence angle.

SUMMARY OF THE INVENTION

In accordance with the present invention, the foregoing objectives are achieved by way of an X-ray Soller slit collimating device that uses lightweight, low density materials that are relatively inexpensive. The density of the material used is less than 6g/cm³. The Soller slit device of the present invention provides an increased transmission throughput efficiency of greater than 60%, while maintaining a low divergence of less than 0.1°.

The present invention, in accordance with an embodiment thereof, provides a Soller slit device whose blades are made of low density materials. Some examples of low density materials used for the blades of the Soller slit of the present invention include glass and mica.

Advantageously, by making Soller slit devices from such low density materials, the blades of the devices of the invention can be made much thinner than traditional Soller slit blades. For example, in accordance with an embodiment of the present invention, glass blades that measure approximately $50\mu\text{m}$ in thickness may be used. Such a thin blade allows for great increases in the throughput efficiency of the Soller slit device. Additionally, the blades of the Soller slit device of the present invention may be spaced with wide spacings therebetween, which also contributes to an increased transmission efficiency.

Additionally, the blades of the Soller slit device of the present invention may be produced in longer lengths, which decreases the angle of divergence of the beam transmitted through the device. These longer lengths are available as the blades of the present invention do not bend like the metal blades of prior devices. In accordance with an embodiment of the present invention, the angle of divergence of the Soller slit device may be less than 0.1° .

Because of the longer length of the blades, lower density materials may be used to construct the blades. This is because a divergent beam, which exceeds the divergence angle of the Soller slit device of the present invention (e.g., greater than 0.1°) strikes the blades of the Soller slit device at an oblique angle that magnifies the absorption capability of each blade by a large factor. For example, in accordance with an embodiment of the present invention, each blade's absorption ability may be multiplied by a factor of about 600. Because of this large absorption factor, glass, mica, and other low density materials provide adequate absorption for divergent high energy radiation, including X-rays.

The present invention also provides for a diffractometer or system for X-ray, or other high radiation, diffractometry which makes use of the Soller slit described above. Specifically, the system for diffractometry provided by the present invention allows for the use of a Soller slit as a collimation element within the system. The Soller slit provides a greatly reduced divergence, and an advantageously increased transmission efficiency. Specifically, transmission efficiency of the Soller slit used by the system for diffractometry allows for a transmission efficiency of at least 60% and preferably approximately 80%, while maintaining a divergence of less than 0.1° . This is accomplished by using a Soller slit manufactured from relatively low density materials.

The foregoing features of the invention, and the advantages achieved thereby, are explained in greater detail hereinafter with reference to particular embodiments illustrated in the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a block diagram of a Soller slit collimator in accordance with an embodiment of the present invention.

Figure 2 is a block diagram of a basic diffractometry system in which the present invention may be used.

DETAILED DESCRIPTION OF THE INVENTION

To facilitate an understanding of the principles that underlie the present invention, it will be described hereinafter with particular reference to embodiments thereof, and specific applications wherein it is used. It will be appreciated by those skilled in the art, however, that the practical applications of the invention are not limited to the particular embodiments described herein. Rather, the invention will find utility in a variety of different applications wherein a Soller slit X-ray collimator having a high transmission throughput efficiency and/or a low divergence associated therewith is desirable. The present invention provides commercial advantages for multiple applications, as the Soller slit device of the present invention provides a transmission efficiency that is much greater and a divergence angle that is much less than those associated with traditional high energy radiation or X-ray optics used in high energy radiation applications, such as X-ray diffractometry.

Figure 1 illustrates a typical Soller slit configuration, which can be modified to form an embodiment of the present invention. In Figure 1, a Soller slit device 100 is shown. The Soller slit device is made up of multiple parallel blades 102a, 102b, 102c, 102d, 102e, 102f. Although only a limited number of blades are shown in Figure 1, it will be appreciated by those skilled in the art that the Soller slit device 100 could be made up of numerous blades, all of which are not shown in the Figure. To the left of the Soller slit device are shown several X-rays, represented by four long arrows. The Soller slit X-ray collimator device 100 works by preventing the divergent X-rays (e.g., X-ray radiating in the direction of the top two illustrated X-rays) from passing through the Soller slit, while allowing the non-diverging X-rays (e.g., the bottom-most X-ray illustrated) to pass through the collimating device 100. Thus, multiple X-rays may be incident on the Soller slit device 100, and only those that are

parallel, or nearly parallel with (i.e., slightly divergent from) the blades of the Soller slit device will be allowed to pass through. All divergent X-rays, on the other hand, will be absorbed by the blades 102.

The key performance parameters of a Soller slit device 100 are its divergence and transmission efficiency. Theoretical divergence of any Soller slit device 100 is given by Equation 1 below:

$$\Delta\Theta = \frac{2d}{l}$$

where $\Delta\Theta$ is the theoretical divergence angle of the Soller slit device 100, d is the spacing between blades 102 of the Soller slit device 100, and l is the length of each blade 102 of the Soller slit device 100. It should be noted, however, that the divergence described in Equation 1 is only theoretical, and that divergence may be worse for Soller slit devices that have manufacturing defects. Thus, a Soller slit device having blades that are not correctly spaced, or properly aligned, for example, may have a divergence that is greater (i.e., worse for most applications) than the divergence calculated by way of Equation 1 above.

Transmission efficiency can be calculated according to Equation 2, shown below:

$$T = \frac{d}{d + t}$$

where T represents transmission efficiency of the Soller slit device 100, d is the distance between the blades 102 of the Soller slit device 100 and t is the thickness of each blade 102 of the Soller slit device. As with the theoretical divergence defined by Equation 1 above, the transmission efficiency defined in Equation 2 is only theoretical, and may be greatly influenced by manufacturing defects. For example, blades that are not perfectly flat or which bend, or blades that do not properly absorb divergent X-rays may reduce the overall transmission efficiency of a Soller slit device.

It should be noted with respect to Equations 1 and 2 that material considerations do not form a part of these equations. However, both of these Equations assume that the materials used for the blades 102 of the Soller slit device 100 provide adequate X-ray absorption, sufficient to prevent divergent X-rays from reflecting, or otherwise passing through the Soller slit device.

To ensure that blades of Soller slit devices are able to absorb X-rays, the blades have traditionally been made of sheets of heavy or highly absorbing metal. Some metals typically used in the construction of Soller slit devices include molybdenum (Mo) or brass. Although metal sheets can be made extremely thin, the mechanical stability of very thin metal sheets is generally not good enough for high precision X-ray work. This is because any curling or rumpling of the sheets (which are common occurrences) will reduce the transmission efficiency as defined by Equation 2 above and yield unpredictable divergence (as defined by Equation 1 above).

It can be seen in Equation 2 that as the thickness of each of the blades 102 of the Soller slit device 100 increases, the transmission efficiency decreases because more of the X-ray radiation within the acceptable angle of divergence (i.e., nearly parallel to the blades) will be absorbed by the blades' edges. Thus, metal-foil blades for Soller slit devices, which have been made with relatively thick foils (e.g., about 250 μ m) produce a low transmission efficiency that worsens further as the required divergence is reduced. Similar problems exist with metal blades, which although they can be made thin, cannot be controlled at thicknesses necessary to produce high transmission efficiencies, as defined by Equation 2 above. Additionally, blades constructed of metal typically have lengths on the order of 3 to 4 cm to prevent bending, which increases the theoretical divergence of the device according to Equation 1.

The present invention, on the other hand, makes use of blades 102 within a modified Soller slit device 100 that are made from low-density material, such as glass, mica, or the like. The density of the material is typically less than 6g/cm³. Advantageously, glass, and the other materials of the present invention from which the blades 102 are made, can be formed in very thin, long sheets that are stable. For example, glass can be formed into sheets having a thickness of only about 50 μ m. These sheets do not bend as is the case with the metal sheets traditionally used to form blades of prior Soller slit devices. Because the glass blades do not bend, the length of the blades 102 of the Soller slit device 100 can be greater than 5 cm, and can be increased to lengths in the range of 12 to 15 cm, or about four to five times the length of traditional blades made from metals.

Therefore, because of the increased length of the blades 102 of the Soller slit device 100 of the present invention, the divergence, as described by Equation 1 above, is greatly reduced. For example, divergences of less than 0.1° are easily obtainable by way of the present invention. In accordance with an embodiment of the present invention, a divergence of 0.07° is possible. This provides great commercial advantage over standard Soller slit devices 100 that produce typical divergence angles of 0.2° to 0.8° .

Additionally, because of the thin profile (t) of the blades 102 of the Soller slit device 100 of the present invention, the transmission efficiency is greatly improved, as can be seen from Equation 2 above. For example, a transmission efficiency of greater than 80% is obtainable by way of the present invention. In accordance with an embodiment of the present invention, utilizing glass blades 102, a transmission efficiency of 60% is easily obtained, while traditional Soller slit devices utilizing metal blades typically produce a transmission efficiency of 30% or less.

The term "glass," as used in connection with the embodiments of the present invention, includes materials that are solid whose atoms do not adopt a crystalline lattice, but which nevertheless cannot easily move past one another. Most types of glass used in connection with embodiments of the present invention are based on silica (SiO_2), generally found in sand. Ingredients are often added to silica to lower the softening temperature from about 1200°C to a temperature convenient to work with. Some additives that are commonly added to silica-based glass include sodium (Na_2O) and calcium (CaO). Additionally, soda-lime glass, which is commonly used for windows and bottles, and is easily formed and shaped, may be used to form the blades of a Soller slit device. When higher strength at high temperatures, a low coefficient of expansion, or good thermal shock resistance is necessary, Borosilicate glass, such as Pyrex, may be used to form the blades of a Soller slit device.

Prior to the invention, as pointed out above, it was thought that the blades 102 of a Soller slit device 100 needed to be made from a high density material, such as heavy metals. Therefore, no Soller slit devices were made by forming blades from low density materials. However, as can be seen with reference to Equation 1 above, when Soller slit devices have relatively large spacings between blades 102 (d), the angle of divergence ($\Delta\Theta$) is greatly reduced by the blades having long lengths (l). It is the combination of long lengths (l), thin non-reflecting blades and relatively large spacings that give the present invention its unique properties. It

has not been found possible to realise these using conventional metallic foils but the combination can be realised by the use of low density materials as described herein. As stated above, the divergence of the Soller slit devices 100 of the present invention is less than 0.1°. Therefore, the length of the oblique path through the Soller slit blades 102 for divergent X-rays must be defined by Equation 3 below:

$$P \geq \frac{t}{\sin(\Delta\Theta)}$$

where P represents the oblique path through the blade 102, in essence the effective thickness for low angle incident X-rays, t represents the thickness of each blade 102, and $\Delta\Theta$ is the divergence of the Soller slit device 100. Thus, for the Soller slit device 100 of the present invention that has a divergence of approximately 0.1° or less, the effective thickness is increased by a factor of approximately 600 compared to the blade thickness (t). Both calculations and experiments have shown that for such Soller slit devices, glass and other low density materials are entirely adequate absorbers of X-ray radiation.

The blades 102 of the Soller slit device 100 should be non-reflective, but the thin glass blades, used in accordance with an embodiment of the present invention to form the blades 102 of the Soller slit device 100 of the present invention, are naturally reflective to most radiation, including high energy radiation, such as X-rays, and EUV radiation. However, this can be easily modified by applying a non-reflective coating, or by etching the surface of the blades. In coating thin glass blades, a number of metals, which have naturally high roughness, can be evaporated onto the glass surface. Some such metals that could be used to form a non-reflective coating on glass blades include gold and platinum, among others. In accordance with an embodiment of the present invention, the coating could be formed from Barium Sulphate (BaSO_4), which is advantageous as it can be formed into a stable, reliable coating having a thickness of only 10-15 μm . Gold, platinum, tungsten, and Barium Sulphate are advantageous as they are also non-corrosive in the atmosphere, and thus can be used for a long period without need for replacement due to corrosion. Additionally, gold, platinum, and Barium Sulphate are relatively dense materials that add to the absorption capability of each of the blades. However, no additional heavy materials are required for absorption purposes to coat the blades 102. Thus, non-reflective coatings could be made of other elements that would suitably prevent reflection of X-rays from the glass blades 102.

Such a non-reflective coating could, for example, include an etched surface of the glass blades.

In accordance with an embodiment of the present invention, the Soller slit device 100 could comprise a number of glass blades 102 having a thickness of 70 μm or less, with a surface coating of 0.5 - 1.0 μm of gold or tungsten. The glass blades 102 of the Soller slit device 100 of the present invention may use precision lapped slips of glass as spacers to control the blade separation. The spacing of the glass blades is critical, and must be maintained to an almost exact precision to prevent from adversely effecting the divergence or transmission efficiency parameters defined by Equations 1 and 2 above.

The Soller slit device 100 of the present invention may be used as the optical element of a high energy radiation imaging system, such as an X-ray diffractometry system. In Figure 2, a block diagram of a basic X-ray diffractometry system 200 is shown in which the present invention may be used. Although Figure 2 relates to a basic X-ray diffractometry system, the basic setup and components associated therewith would also be associated with other diffractometry systems using different forms of high energy radiation. Therefore, any discussion of the implementation of the Soller slit collimator within the X-ray diffractometry system of Figure 2 can also be applied to other high energy radiation diffractometry systems.

The diffractometry system 200 of Figure 2 utilizes an X-ray line source 202 to produce the X-rays used to analyze a sample. This source 202 may comprise, for example, a laser beam vaporizing metal foil, such as copper foil, which creates multiple charged ions that emit X-ray radiation. The line source 202 shown in Figure 2 is perpendicular to the plane of the paper in which Figure 2 is shown.

X-ray radiation from the source 202 passes through a vertical divergence control unit 204. This vertical divergence control unit 204 is typically a group of axial Soller slits. These Soller slits are parallel to the plane of the paper in which Figure 2 is shown, and are not low-divergence Soller slits. It will be recognized by those skilled in the art that the Soller slit device of the present invention could be used as the vertical divergence control unit 204. However, as low-divergence is not necessary, the high quality and low-divergence associated with the Soller slit device of the present invention are not required.

After passing through the vertical divergence control unit 204, X-ray radiation then passes through incident beam divergence slits 206, which serve as slit apertures. After passing through the incident beam divergence slits 206, the X-ray radiation impinges on the specimen 208. It is this specimen 208 that is being examined by way of the X-ray diffractometry system shown in Figure 2. The specimen then diffracts the incident X-ray radiation, creating diffracted X-ray radiation, which then passes through X-ray collimating optics 210.

In accordance with an embodiment of the present invention, the X-ray collimating optics 210 may make use of the Soller slit device described herein having blades made of low density materials. The blades of the Soller slit device 210 are shown as being perpendicular to the plane of the drawing of Figure 2. As described above, the Soller slit device 210 has the advantageous effect of producing low-divergence X-ray radiation with an angle of divergence of 0.1° or less, and with a high transmission efficiency of approximately 80%.

Once the diffracted X-ray radiation has been collimated by way of the Soller slit device 210, it is then reflected off a monochromator crystal 212 to the detector 214. The monochromator crystal 212 serves to isolate the desired wavelength of the diffracted X-ray by diffraction, and may be any suitable monochromator crystal, including graphite, for example. The detector 214 may comprise any detector suitable for detecting diffracted X-ray radiation. An embodiment of the present invention, for example, makes use of a scintillation detector.

The present invention, when utilized as the collimating optics 210, exhibits distinct advantages over traditional high energy radiation optics in that it improves both peak width and peak intensity of diffraction patterns produced by a high energy (e.g., X-ray or EUV) diffractometer. First, the present invention provides narrower diffraction peak widths when used in an X-ray diffractometer, which is commercially advantageous. Generally, the width of peaks measured on a diffractometer depends on two parameters: sample quality and instrument broadening. Sample quality includes such factors as sample disorder, thermal vibration, particle size, strain/stress within the lattice, and the quality of the alignment of the diffracting planes with the crystal lattice. These factors are generally not improved by way of improved X-ray optics. However, instrument broadening is controlled mainly by the X-ray optics, and specifically a Soller slit device such as the present invention, when used within an X-ray diffractometry system. Thus, instrument broadening, which contributes to increased peak width, can be minimized by using an efficient and effective Soller slit.

X-rays are naturally divergent. Diffractometers are designed to guide X-rays from the source into the sample and then into the detector to measure scattered intensities as a function of a scattering angle. The X-ray beam is directed by optical elements, the most basic one being slits, or more sophisticated multi-layer optics, or Soller slits. Thus, the use of X-ray optics can control the beam divergence, but can rarely ever eliminate divergence entirely. The present invention, which provides a minimum angle of divergence, decreases the overall instrument broadening and thus contributes to a narrower peak width.

The measured peak width of diffraction patterns produced by X-ray diffractometry is a combination of sample quality broadening and instrument broadening. More specifically, the measured peak width is generally a convolution of both broadening effects. Thus, the worse the sample quality and the larger the beam divergence, or instrument broadening, the broader the measured peak becomes. Therefore, instrument broadening essentially sets a lower limit of peak width that theoretically can be measured on a particular instrument. The present invention provides minimum instrument broadening, thereby approaching the lower limit of peak width that theoretically can be measured by a given diffractometer. Narrow diffraction peak widths are desired to increase resolution of similar constituents of a sample. Thus, by way of the present invention, similar constituents that may not be able to be independently resolved on instruments using optics that produce a greater divergence angle (i.e., instruments with greater instrument broadening) can be independently resolved by way of embodiments of the present invention. That is, by maintaining a narrower peak resolution, embodiments of the present invention are able to obtain more information in a given sample.

The second parameter that contributes to the performance of X-ray diffractometers is peak intensity. In X-ray diffractometry, because of the nature of the X-ray radiation used, it is difficult to obtain an increase in peak intensity. As mentioned above, it is typical to analyze a sample over a relatively long period of time to collect a large amount of data from which noise may be subtracted, such as thermal noise, and the like. This technique helps the observed peak intensity to increase. However, if peak intensity is improved, then less time is required to collect adequate measurements of a particular sample. For example, in X-ray diffractometry, an improvement by a factor of two of the peak intensity is extremely important, as peak intensities are low, and generally near the ambient noise floor.

Some of the factors that influence peak intensity include: intensity of the primary beam, sample absorption, and efficiency of the X-ray optics. The transmission efficiency of the X-ray optics can be greatly improved by utilizing the Soller slit device 100 of the present invention. For example, the increased transmission efficiency of the Soller slit of the present invention greatly contributes to peak intensity in an X-ray diffractometry application.

Thus, by way of the foregoing, it can be seen that the Soller slit of the present invention provides a tremendous commercial advantage, as it is able to produce narrow and intense diffraction peaks for X-ray diffractometry and similar high energy radiation applications. Specifically, the Soller slit of the present invention, when used as part of the X-ray optics of an X-ray diffractometry system, is able to increase peak intensity and reduce instrument broadening, which consequently narrows measured peak widths. More generally, the present invention provides for a Soller slit device for use with high energy radiation, such as X-ray or EUV radiation that minimizes divergence and increases transmission efficiency.

It will be appreciated by those of ordinary skill in the art that the present invention can be embodied in other specific forms without departing from the scope or essential characteristics thereof. For example, while an exemplary embodiment of the present invention has been described with reference to a Soller slit for X-ray collimation, the principles of the present invention are applicable to collimation of other radiation that behaves similarly to that of X-rays, such as extreme ultraviolet (EUV) and other types of high energy radiation.

Additionally, although the present invention has been described in connection with its use and applicability within an X-ray diffractometry system, it will be appreciated by those skilled in the art that the Soller slit device of the present invention can be usefully employed in any system where collimation of X-ray or other similarly behaving radiation is required and/or desired.

The presently disclosed embodiments are, therefore, considered in all respects to be illustrative, and not restrictive. The scope of the invention is indicated by the appended claims, rather than the foregoing description, and all changes that come within the meaning and range of equivalents thereof are intended to be embraced therein.